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U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

Technical News

BULLETIN

VOLUME 47 OCTOBER 1963 NUMBER 10

U.S. DEPARTMENT OF COMMERCE — LUTHER H. HODGES, *Secretary*

NATIONAL BUREAU OF STANDARDS — A. V. ASTIN, *Director*

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COVER: Tungsten-spring apparatus used to determine the rate of degradation of polytrifluoroethylene previously exposed to gamma irradiation. Information on the behavior of materials exposed to high-energy radiation should be useful in estimating their resistance to outer space environments. Sidney Straus uses a cathetometer to observe the deflection of a crossline on the tungsten spring (enclosed in evacuated glass system at right). As the sample (suspended from the spring in a small bucket) is heated at a constant temperature, it decomposes and loses weight, resulting in a change in the position of the crossline (see p. 189).

New Values for the Physical Constants

Recommended by NAS–NRC

A NEW, consistent set of values for the physical constants has been recommended by the Committee on Fundamental Constants of the National Academy of Sciences–National Research Council. The work of this committee is a continuation of the work of an earlier NAS–NRC committee.

The National Bureau of Standards has adopted these values [1] for use in all NBS publications, except where for some special reason the author is required to use others. No doubt these constants will have additional wide use. It is anticipated that the International Union of Pure and Applied Chemistry and the International Union of Pure and Applied Physics will encourage their employment on an international basis.

Previous to this action of the NAS–NRC Committee, several lists of "best values" for the physical constants were available. The current issue of the Smithsonian physical tables, for example, lists three separate sets of values. The individual research worker is then forced to make a choice as to which list he is going to follow.

Uniformity and consistency in the use of such values are necessary for an adequate exchange of information among scientists. For example, the Faraday constant is given by $F=N_A e$. Different values for all three of these constants appear in the various tables, but in order to satisfy the equation it is necessary that all values be taken from the same consistent set.

The members of the NAS–NRC Committee are J. A. Bearden, E. R. Cohen, J. W. M. DuMond, L. P. McCullough, N. Ramsey, F. D. Rossini, J. S. Thomsen, G. Waddington, and Alvin G. McNish, *Chairman*. Many of these individuals have been very active in the development of consistent sets of constants over the past few years.

The Committee reports: "In arriving at these values the Committee had available a large amount of experimental information not all of which appeared to be of consistent reliability and accuracy. In some cases where experiments were suspected of having significant errors and the results were discrepant with other experimental results believed to be of greater reliability, the data were rejected. Inclusion of these rejected data would have altered the values in the table somewhat but not greatly."

The results of several recent experimental researches of the National Bureau of Standards have been of great importance in establishing the new values. These were the new determinations of the gyromagnetic ratio, of the electrochemical equivalent of silver, the relative abundances of the isotopes of silver, and the value of the absolute ampere. A full report of the calculations and considerations entering into this new set of values was discussed by J. W. M. DuMond and E. R. Cohen at the Second International Conference on Nuclidic Masses in Vienna, in July 1963 (to be published in the Conference Proceedings).

The new values of the constants are given in table 2. The uncertainties assigned represent established limits of error; namely, three standard errors based on standard deviations assigned to the data retained for input to the least-squares adjustment. Those calculated from the residuals of the final least-square adjustment are even smaller. It is therefore unlikely that the true value of any of the constants differs from the value given in the table by as much as the stated uncertainty. The uncertainties in constants computed from elemental ones may be either greater or less than is obtained by a simple combination of the elemental

TABLE 1. *Defined values and equivalents*

Meter	(m)	1 650 763.73 wavelengths in vacuo of the unperturbed transition $2p_{10}-5d_5$ in ^{86}Kr
Kilogram	(kg)	mass of the international kilogram at Sèvres, France
Second	(s)	1/31 556 925.974 7 of the tropical year at 12^h ET, 0 January 1900
Degree Kelvin	(°K)	defined in the thermodynamic scale by assigning 273.16 °K to the triple point of water (freezing point, 273.15 °K=0 °C)
Unified atomic mass unit	(*u*)	1/12 the mass of an atom of the ^{12}C nuclide
Mole	(mol)	amount of substance containing the same number of atoms as 12g of pure ^{12}C
Standard acceleration of free fall	(g_n)	9.806 65 m s^{-2}, 980.665 cm s^{-2}
Normal atmospheric pressure	(atm)	101 325 N m^{-2}, 1 013 250 dyn cm^{-2}
Thermochemical calorie	(cal_{th})	4.1840 J, 4.1840×10^7 erg
International Steam Table calorie	(cal_{IT})	4.1868 J, 4.1868×10^7 erg
Liter	(l)	0.001 000 028 m^3, 1 000.028 cm^3 (recommended by CIPM, 1950)
Inch	(in.)	0.0254 m, 2.54 cm
Pound (avdp.)	(lb)	0.453 592 37 kg, 453.592 37 g

TABLE 2. *Adjusted values of constants*

Constant	Symbol	Value	Est. ‡ error limit	Unit			
				Système International (MKSA)		Centimeter-gram-second (CGS)	
Speed of light in vacuum	c	2.997925	3	$\times 10^{8}$	m s^{-1}	$\times 10^{10}$	cm s^{-1}
Elementary charge	e	1.60210	7	10^{-19}	C	10^{-20}	$cm^{1/2}g^{1/2}$ *
		4.80298	20	.....	.....	10^{-10}	$cm^{3/2}g^{1/2}s^{-1}$ †
Avogadro constant	N_A	6.02252	28	10^{23}	mol^{-1}	10^{23}	mol^{-1}
Electron rest mass	m_e	9.1091	4	10^{-31}	kg	10^{-28}	g
		5.48597	9	10^{-4}	u	10^{-4}	u
Proton rest mass	m_p	1.67252	8	10^{-27}	kg	10^{-24}	g
		1.00727663	24	10^{0}	u	10^{0}	u
Neutron rest mass	m_n	1.67482	8	10^{-27}	kg	10^{-24}	g
		1.0086654	13	10^{0}	u	10^{0}	u
Faraday constant	F	9.64870	16	10^{4}	C mol^{-1}	10^{3}	$cm^{1/2}g^{1/2}mol^{-1}$ *
		2.89261	5	.....	.....	10^{14}	$cm^{3/2}g^{1/2}s^{-1}mol^{-1}$ †
Planck constant	h	6.6256	5	10^{-34}	J s	10^{-27}	erg s
	$\hbar$	1.05450	7	10^{-34}	J s	10^{-27}	erg s
Fine structure constant	α	7.29720	10	10^{-3}	.....	10^{-3}	
	$1/\alpha$	1.370388	19	10^{2}	.....	10^{2}	
	$\alpha/2\pi$	1.161385	16	10^{-3}	.....	10^{-3}	
	α^2	5.32492	14	10^{-5}	.....	10^{-5}	
Charge to mass ratio for electron	e/m_e	1.758796	19	10^{11}	C kg^{-1}	10^{7}	$cm^{1/2}g^{-1/2}$ *
		5.27274	6	.....	.....	10^{17}	$cm^{3/2}g^{-1/2}s^{-1}$ †
Quantum-charge ratio	h/e	4.13556	12	10^{-15}	J s C^{-1}	10^{-7}	$cm^{3/2}g^{1/2}s^{-1}$ *
		1.37947	4	.....	.....	10^{-17}	$cm^{1/2}g^{1/2}$ †
Compton wavelength of electron	λ_C	2.42621	6	10^{-12}	m	10^{-10}	cm
	$\lambda_C/2\pi$	3.86144	9	10^{-13}	m	10^{-11}	cm
Compton wavelength of proton	$\lambda_{C,p}$	1.32140	4	10^{-15}	m	10^{-13}	cm
	$\lambda_{C,p}/2\pi$	2.10307	6	10^{-16}	m	10^{-14}	cm
Rydberg constant	R_∞	1.0973731	3	10^{7}	m^{-1}	10^{5}	cm^{-1}
Bohr radius	a_0	5.29167	7	10^{-11}	m	10^{-9}	cm
Electron radius	r_e	2.81777	11	10^{-15}	m	10^{-13}	cm
	r_e^2	7.9398	6	10^{-30}	m^2	10^{-26}	cm^2
Thomson cross section	$8\pi r_e^2/3$	6.6516	5	10^{-29}	m^2	10^{-25}	cm^2
Gyromagnetic ratio of proton	γ	2.67519	2	10^{8}	rad $s^{-1}T^{-1}$	10^{4}	rad $s^{-1}G^{-1}$ *
	$\gamma/2\pi$	4.25770	3	10^{7}	Hz T^{-1}	10^{3}	$s^{-1}G^{-1}$ *
(uncorrected for diamagnetism, H_2O)	γ'	2.67512	2	10^{8}	rad $s^{-1}T^{-1}$	10^{4}	rad $s^{-1}G^{-1}$ *
	$\gamma'/2\pi$	4.25759	3	10^{7}	Hz T^{-1}	10^{3}	$s^{-1}G^{-1}$ *
Bohr magneton	μ_B	9.2732	6	10^{-24}	J T^{-1}	10^{-21}	erg G^{-1} *
Nuclear magneton	μ_N	5.0505	4	10^{-27}	J T^{-1}	10^{-24}	erg G^{-1} *
Proton moment	μ_p	1.41049	13	10^{-26}	J T^{-1}	10^{-23}	erg G^{-1} *
	μ_p/μ_N	2.79276	7	10^{0}	.....	10^{0}	
(uncorrected for diamagnetism, H_2O)	μ'_p/μ_N	2.79268	7	10^{0}	.....	10^{0}	
Anomalous electron moment corrn.	$(\mu_e/\mu_0)-1$	1.159615	15	10^{-3}	.....	10^{-3}	
Zeeman splitting constant	μ_B/hc	4.66858	4	10^{1}	$m^{-1}T^{-1}$	10^{-5}	$cm^{-1}G^{-1}$ *
Gas constant	R	8.3143	12	10^{0}	J $°K^{-1}$ mol^{-1}	10^{7}	erg $°K^{-1}$ mol^{-1}
Normal volume perfect gas	V_0	2.24136	30	10^{-2}	m^3 mol^{-1}	10^{4}	cm^3 mol^{-1}
Boltzmann constant	k	1.38054	18	10^{-23}	J $°K^{-1}$	10^{-16}	erg $°K^{-1}$
First radiation constant $(2\pi hc^2)$	c_1	3.7405	3	10^{-8}	W m^2	10^{-5}	erg cm^2 s^{-1}
Second radiation constant	c_2	1.43879	19	10^{-2}	m °K	10^{0}	cm °K
Wien displacement constant	b	2.8978	4	10^{-3}	m °K	10^{-1}	cm °K
Stefan-Boltzmann constant	σ	5.6697	29	10^{-8}	W m^{-2} $°K^{-4}$	10^{-5}	erg cm^{-2} s^{-1} $°K^{-4}$
Gravitational constant	G	6.670	15	10^{-11}	N m^2 kg^{-2}	10^{-8}	dyn cm^2 g^{-2}

‡Based on 3 std. dev.; applied to last digits in preceding column. *Electromagnetic system. †Electrostatic system.
C—coulomb J—joule Hz—hertz W—watt N—newton T—tesla G—gauss

TABLE 3. *Energy conversion factors*

	Formula	Factor	Error limit	Conversion			
				Système International (MKSA)		Centimeter-gram-second (CGS)	
Electron-volt	eV	1.60210	7	$\times 10^{-19}$	$J(eV)^{-1}$	$\times 10^{-12}$	$erg\ (eV)^{-1}$
Energy associated with							
Unified atomic mass unit	c^2/Ne	9.31478	15	10^8	$eV\ u^{-1}$	10^8	$eV\ u^{-1}$
Proton mass	$m_p c^2/e$	9.38256	15	10^8	$eV\ m_p^{-1}$	10^8	$eV\ m_p^{-1}$
Neutron mass	$m_n c^2/e$	9.39550	15	10^8	$eV\ m_n^{-1}$	10^8	$eV\ m_n^{-1}$
Electron mass	$m_e c^2/e$	5.11006	5	10^5	$eV\ m_e^{-1}$	10^5	$eV\ m_e^{-1}$
Cycle	e/h	2.41804	7	10^{14}	$Hz(eV)^{-1}$	10^{14}	$s^{-1}(eV)^{-1}$
Wavelength	ch/e	1.23981	4	10^{-6}	$eV\ m$	10^{-4}	$eV\ cm$
Wave number	e/ch	8.06573	23	10^3	$m^{-1}(eV)^{-1}$	10^1	$cm^{-1}(eV)^{-1}$
°K	e/k	1.16049	16	10^4	$°K(eV)^{-1}$	10^4	$°K(eV)^{-1}$

uncertainties because of the correlation among the elemental constants introduced by the least-squares adjustment (see ref. DuMond and Cohen). The values are based upon the assumption that the acceleration of gravity is correctly given by adding −0.013 gal to the value of g given in the Potsdam system. All the values where pertinent are referred to the unified system of atomic masses in which the atomic mass of the atom of the nuclide ^{12}C is assigned the numerical value 12 exactly. Symbols recommended by the Commission on Symbols, Units, and Nomenclature (SUN Commission) of the International Union of Pure and Applied Physics are employed. (See June 1962 *Physics Today*.)

[1] *A wallet-size card presenting recommended values of selected physical constants may be obtained upon request from the Office of Technical Information, National Bureau of Standards, Washington, D.C. 20234.*

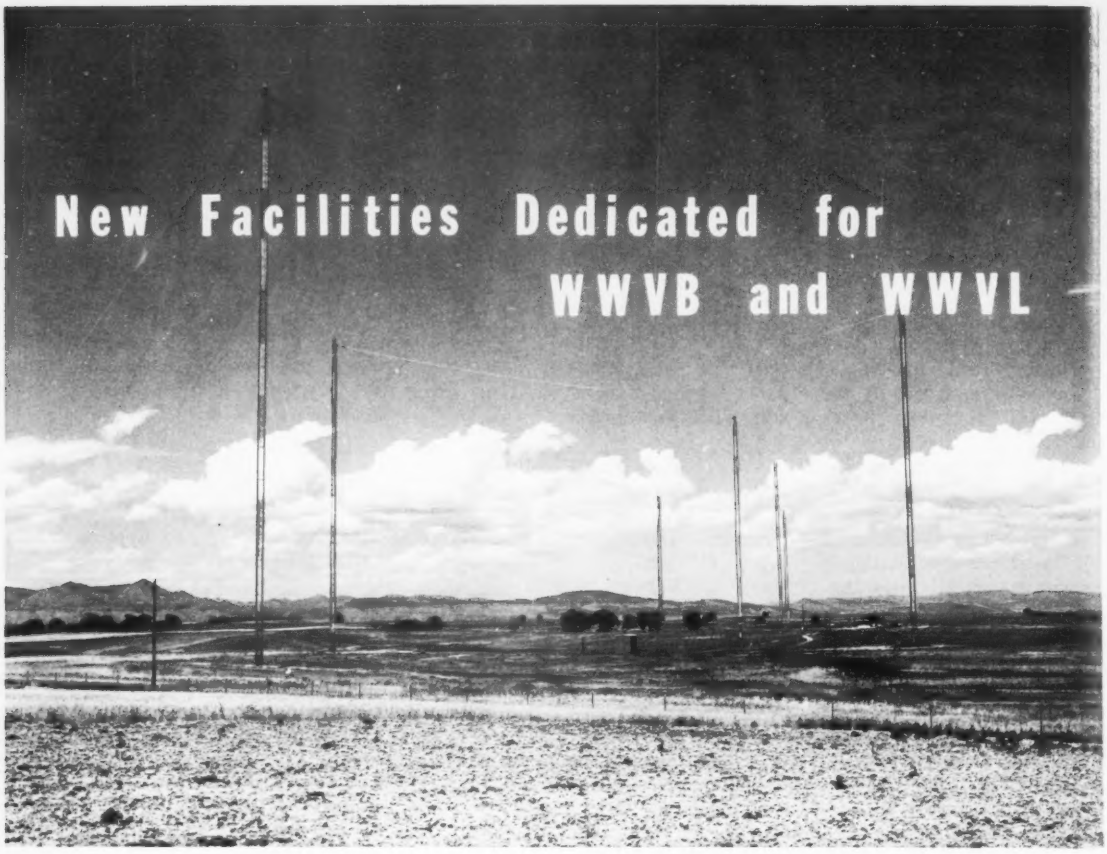
Change in Broadcast Schedule of WWVH

On October 1, 1963, NBS radio station WWVH, Maui, Hawaii, eliminated the present 34-minute silent period at 1900 UT daily. The silent period from 15 to 19 minutes past each hour is being continued.

WWVH is one of four stations operated by NBS to provide standard frequency and time signals. The signals, referred to the United States Frequency Standard, are widely used by radio and television stations, electric power companies, electronics manufacturers, calibration laboratories, and space and missile installations. The following table lists the major characteristics of the stations.

	WWV [1]	WWVH [1]	WWVB [2]	WWVL [2]
Location	Beltsville, Md.	Maui, Hawaii	Ft. Collins, Colo.	Ft. Collins, Colo.
Frequencies	2.5, 5, 10, 15, 20, 25 Mc/s.	5, 10, 15 Mc/s	60 kc/s	20 kc/s
Services				
Standard radio frequencies	Yes	Yes	Yes	Yes
Time signals	Yes	Yes	Yes	No [3]
Standard audio frequencies	Yes	Yes	No	No
Standard musical pitch	Yes	Yes	No	No
Radio propagation forecasts	Yes	Yes	No	No
Geophysical alerts	Yes	Yes	No	No

[1] See NBS Misc. Publ. 236 or June 1963 ISA Journal, for additional information.
[2] See p. 178 of this issue.
[3] To be added.



NEW facilities, providing greatly increased power and range, were dedicated for standard broadcast stations WWVB and WWVL on August 13, 1963. Operated by the NBS Boulder Laboratories at a site near Fort Collins, Colo., these stations transmit standard frequencies which are received at greater accuracy than those of NBS high-frequency stations WWV and WWVH. This higher accuracy is required in many satellite and missile programs and for basic research on atmospheric and ionospheric phenomena.

Until recently, stations WWVB and WWVL have been used only for experimental low-frequency transmissions—WWVB broadcasting at 60 kc/s from Boulder, Colo., and WWVL at 20 kc/s from Sunset, Colo. The success of the experimental broadcasts provided technical justification for the construction of new facilities at Fort Collins and establishment of the two stations on a permanent basis. The new stations complement but do not replace WWV and WWVH, which are sufficiently accurate for many important applications.

Special dedication ceremonies for the low-frequency facilities were held at the NBS Boulder Laboratories during the Symposium on Ionospheric Propagation of Very Low Frequency Waves (August 12–14). Dr. J. M. Richardson, Chief of the NBS Radio Standards Laboratory, was chairman of the dedication session, at which Dr. A. V. Astin, NBS Director, and Dr. R. D. Huntoon, Deputy Director for Basic Standards and Services, spoke. The dedication was followed by a technical session devoted to the new stations and their use, after which the Symposium delegates had an opportunity to visit the Fort Collins site.

The Bureau has for many years provided standard frequency and time broadcasts over high-frequency stations WWV and WWVH. These high-frequency signals are propagated over long distances by alternate reflections between the earth and the ionosphere. As the height and density of the ionosphere change constantly, resulting in changes in the path of the radio waves, there is a loss of accuracy in the signals at the point of reception. To overcome this inherent limitation of high-frequency transmissions, WWVB and WWVL are operated in the low- and very-low-frequency regions, respectively. Their radio waves follow the curvature of the earth, with the ground and the ionosphere acting as the lower and upper limits of a gigantic duct to guide the signals over the globe. As the iono-

Opposite page: **The new facilities for WWVL and WWVB will provide frequency standard broadcasts at 20 kc/s (WWVL) and 60 kc/s (WWVB) with greater received accuracy than was previously available. The antenna for each station consists of four steel towers arranged in a diamond 1900 ft long and 750 ft wide.**

sphere acts as a boundary and not as a reflector, its variations have almost no effect on the travel of the waves; thus the stability of the received signals is increased. The stability of the signals of all Bureau stations is 2 parts in 100 billion at the transmitter.

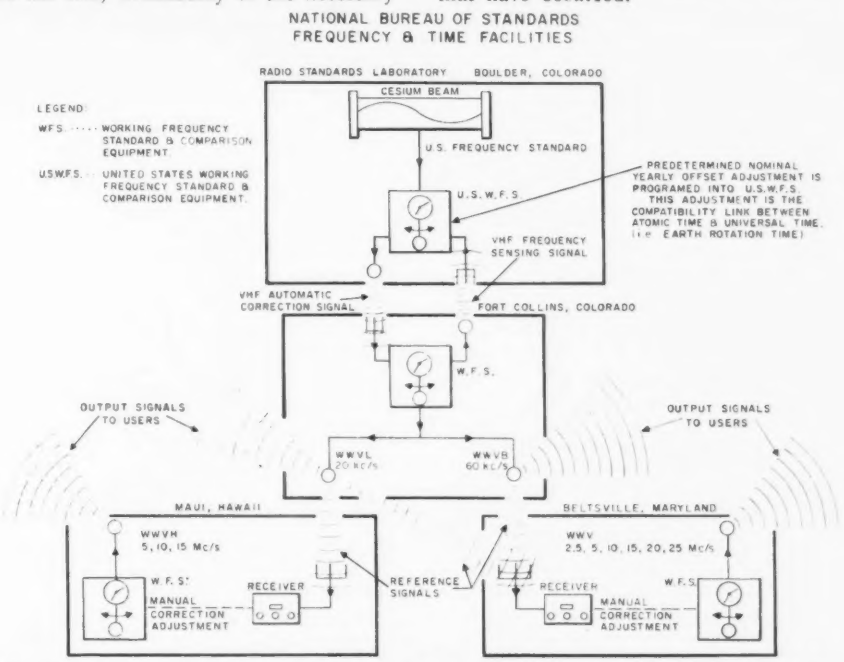
Station WWVB broadcasts at 60 kc/s, with a radiated power of 5 (later to be increased to 7) kilowatts. The signal as received is some 100 times more stable than those available from WWV and WWVH. This station is designed to serve the continental United States, as it provides more stable coverage at distances up to 2,000 miles than does its sister station WWVL. WWVB will be of service to the Air Force, U.S. Geological Survey, geophysical and seismic laboratories, missile ranges, and laboratories concerned with high-precision frequency research and calibration.

WWVL operates at 20 kc/s, with a radiated power of 1 kw. Frequency signals only are now being broadcast; time signals will be added later. The 20-kc/s signal of station WWVL provides intercontinental reception with a precision of 1 part in 10 billion in an observing period of about 1 day. Very-long-distance reception is important to international standardization activities, military bases, and the National Aeronautics and Space Administration. NASA, in fact, partially supported the construction of WWVL.

The Fort Collins site, covering 380 acres, was chosen because of the following advantages: High electrical conductivity of the soil; availability of the necessary electrical power; relative freedom from violent weather; ease of access to the site and nearness to NBS Boulder (not so near, however, as to interfere excessively with Boulder experiments); and a location away from major air routes.

The antenna array for each station consists of four guyed steel towers, arranged in a diamond 1,900 ft long and 750 ft wide. Counterbalances are arranged at the inside and base of each tower to maintain proper tension at the top of the tower and to compensate evenly for winds which may sweep down from the nearby Rocky Mountains. A transmitter building is located between the two antennas; a helix house, used for resonance tuning, terminates each antenna.

A high-quality quartz crystal oscillator at the site provides stable carrier frequencies for the transmitters. To insure that the phase of these frequencies agree with that of the United States Working Frequency Standard maintained at NBS Boulder, a servo loop has been established between the site and the Boulder Laboratories. The carrier phases as received at Boulder are compared with the standard by means of phase detectors, any phase difference resulting in error signals. These error signals, along with a servo motor reference phase, modulate a 50 Mc/s FM telemetering transmitter at Boulder. The FM signals received at Fort Collins are applied to servo motors to correct phase errors that have occurred.


NATIONAL BUREAU OF STANDARDS
FREQUENCY & TIME FACILITIES
RADIO STANDARDS LABORATORY BOULDER, COLORADO
CESIUM BEAM
U.S. FREQUENCY STANDARD
U.S.W.F.S.
PREDETERMINED NOMINAL YEARLY OFFSET ADJUSTMENT IS PROGRAMED INTO U.S.W.F.S. THIS ADJUSTMENT IS THE COMPATIBILITY LINK BETWEEN ATOMIC TIME & UNIVERSAL TIME. (i.e. EARTH ROTATION TIME)
VHF FREQUENCY SENSING SIGNAL
VHF AUTOMATIC CORRECTION SIGNAL
FORT COLLINS, COLORADO
W.F.S.
OUTPUT SIGNALS TO USERS
OUTPUT SIGNALS TO USERS
WWVL 20 KC/S
WWVB 60 KC/S
LEGEND:
W.F.S. ..... WORKING FREQUENCY STANDARD & COMPARISON EQUIPMENT.
U.S.W.F.S. ... UNITED STATES WORKING FREQUENCY STANDARD & COMPARISON EQUIPMENT.
MAUI, HAWAII
WWVH 5, 10, 15 Mc/s
W.F.S.
MANUAL CORRECTION ADJUSTMENT
RECEIVER
REFERENCE SIGNALS
BELTSVILLE, MARYLAND
WWV 2.5, 5, 10, 15, 20, 25 Mc/s
RECEIVER
W.F.S.
MANUAL CORRECTION ADJUSTMENT

Drawing depicting the relationships between the cesium beam frequency standard and the U.S. Working Frequency Standard at NBS Boulder, and the standard frequency and time broadcasts from stations WWVL, WWVB, WWVH, and WWV.

A plaque and portrait photograph were placed at the Fort Collins site of WWVB and WWVL in tribute to W. D. George. Mr. George, killed in an automobile accident while attending an international meeting in Switzerland early this year, devoted his 33-year career at NBS to radio standards and measurement. Here *(left to right)* **Dr. J. M. Richardson, Chief of the NBS Radio Standards Laboratory; Dr. R. D. Huntoon, NBS Deputy Director; Dr. A. V. Astin, NBS Director; Dr. R. B. Scott, Manager, Boulder Laboratories; and Dr. Y. Beers, Chief of the NBS Radio Standards Physics Division, are shown with the portrait.**

Special Tribute to W. D. George

During the dedication ceremonies a bronze plaque, together with a large framed photograph, was placed at the Fort Collins site in memory of William Donovan George. Mr. George, a Consultant to the Chief of the Radio Standards Physics Division, was killed February 12, 1963, in an automobile accident in Switzerland. Mr. George was in Geneva as a U.S. Delegate to the International Radio Consultative Committee (CCIR).

Mr. George, a veteran of 33 years' service with NBS, had a distinguished career in the field of radio and electronics. Joining the Bureau after his graduation from the Georgia Institute of Technology, he devoted himself to the development of radio standards and measurements. In 1946 he was appointed Chief of the High Frequency Standards Section, and later served as Acting Chief of the Radio Standards Division at Boulder. Mr. George, author of 20 technical papers, served at both national and international conferences on radio propagation, and served as Chairman of U.S. Study Group VII—Standard Frequency Transmissions and Time Signals of the CCIR.

Stress-Corrosion Studies of Stainless Steel

data obtained with new type of specimen

A SPECIALLY designed specimen was recently used at the Bureau to investigate the stress-corrosion cracking of metals at elevated temperatures. This specimen, hollowed out so that the corrodent may be sealed into the cavity before stressing begins, was developed by H. L. Logan of the metal reactions laboratory in a study sponsored by the Atomic Energy Commission.[1] Data recently obtained with austenitic stainless-steel specimens of this type may be used to determine the conditions under which the alloy can be utilized safely for reactor components.[2]

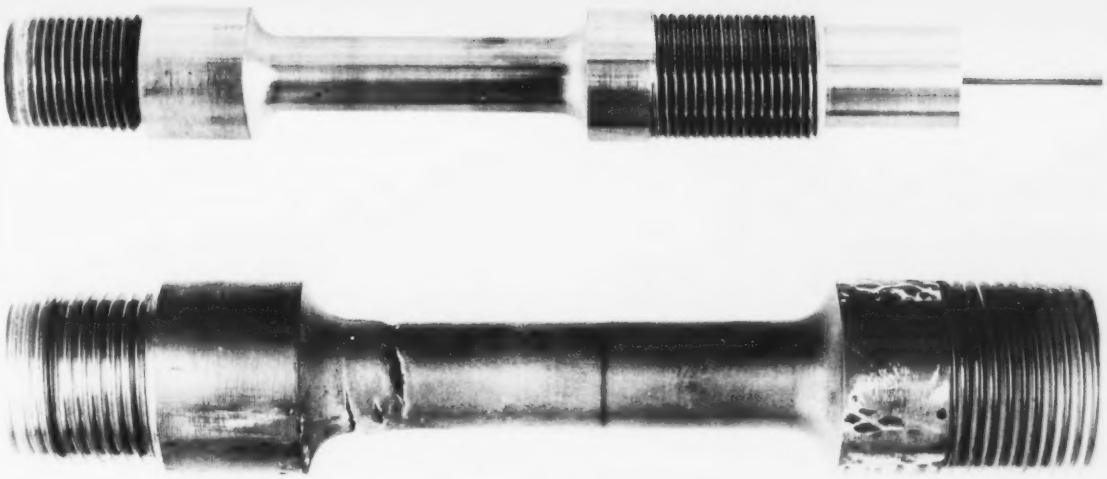
In the method generally employed to evaluate the resistance of metals to stress-corrosion at elevated temperatures, both the specimen and the corrodent are placed in an autoclave in which they may be held at the desired temperature until cracking occurs. To apply stress, the specimen is bent into an arc or a U-shape and restrained from returning to an equilibrium position. However, with this technique no determinations can be made of the threshold value of stress necessary to start cracking nor of the changes that occur in strain with time-at-temperature.

To obtain such data, a specimen was designed that would itself serve as an autoclave which could be heated in a conventional creep furnace and simultaneously subjected to a known tensile stress. Specimens produced according to this design were machined from 1-in.-diam rods into 6 in. lengths. To make the cavity for holding the corrodent, a hole 0.50 in. in diameter was bored to a depth of approximately 4 in. along the longitudinal axis of each specimen. A reduced section about 2 in. in length was machined symmetrically on the outer surface about the longitudinal and transverse axes to achieve wall thicknesses of 0.034 or of 0.060 in. in this section. An austenitic stainless-steel plug was used to close the open end of the cavity, with a tube extending through the plug so that oxygen could be forced into the specimen cavity.

Concentrated Solution Experiments

In one series of experiments, the resistance of type 304 austenitic stainless steel to stress-corrosion cracking was investigated with a corrodent consisting of distilled water containing 20,000 ppm of chloride ion as sodium chloride. After the specimens were sealed and before they were heated, oxygen was forced into the cavities at a pressure of 100 psi. In a number of the specimens, the air above the corrodent in the cavity was also carefully displaced with oxygen at atmospheric pressure before the plug was inserted and the oxygen forced in under pressure.

Of 17 specimens exposed in this way under a stress of 30,000 lb/in.^{2} at preselected temperatures in the range of from 455 to 615 °F, 13 failed completely as a consequence of stress-corrosion cracking, with the



Top: **Specially designed specimen with a hollowed-out reduced section for stress-corrosion experiments. The cavity contains a corrodent and oxygen, when the specimen is heated and stressed in tension. The tube extending from the top of the specimen is used to force the oxygen into the cavity after the specimen is sealed.** *Bottom:* **This specimen failed when internal cracks penetrated the specimen wall.**

cracks penetrating through the cavity wall of the reduced section. Stress-corrosion cracks were found by metallographic examination in the walls of three of the remaining specimens. In this part of the investigation, the combined oxygen treatment usually produced more rapid and severe failures than did the single treatment in which oxygen was forced into the specimen cavity after sealing.

Complete failures were rare in specimens exposed at a stress level of 25,000 psi; however, metallographic examination revealed stress-corrosion cracks penetrating into the interior walls of most of these specimens. Two of the specimens stressed at 25,000 psi and containing no oxygen did not develop any cracks during the exposure period, which was of 300 hr duration, unless earlier failure occurred.

Experiments With Dilute Solutions

In a second series of similar experiments, 34 specimens were exposed to corrodents containing from 100 ppm down to as little as 2 ppm of chloride. In most cases oxygen was also added to specimen cavities. Of two specimens subjected to stresses of 20,000 psi at 570 °F and exposed to a corrodent containing 5 ppm of chloride, one developed stress-corrosion cracks. Cracks were not found in specimens containing 2 ppm of chloride subjected to stresses up to 40,000 psi at 570 °F, nor in those stressed at 15,000 psi and 570 °F and containing 5 or 25 ppm of chloride. No evidence of corrosion was found in a specimen exposed only to a very high purity water at the same temperature, under a stress of 30,000 psi for 4,012 hr, in a high-purity nitrogen atmosphere.

Corrosion Products

At the conclusion of the experiments, the chromate ion, CrO_4^{--}, was found in the dilute solution removed from 24 of these specimens, and the nickel ion, Ni^{++}, was found in 8. A crystalline material that had deposited at the opening of a leak in one of the specimens containing the concentrated solution was identified by X-ray diffraction methods as sodium chromate. Sometimes dark corrosion products covered part of the interior wall of the specimen adjacent to the liquid line. Frequently these products were not continuous but were isolated at pits or at the apparent origins of cracks. X-ray diffraction patterns indicated that these products were primarily gamma ferric oxide.

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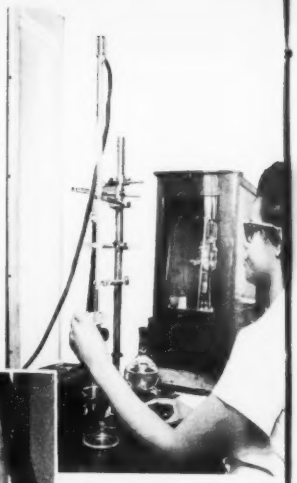
[1] A specimen for use in investigating the stress-corrosion cracking of metals at elevated temperatures, *by H. L. Logan, ASTM Mater. Res. Stds.* **2**, *98 (1962).*

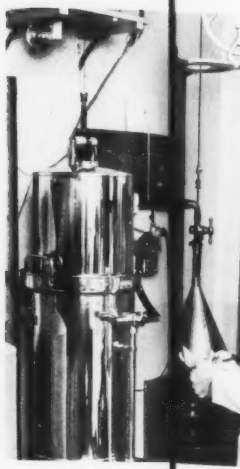
[2] *For further technical details, see* The stress-corrosion cracking of type 304 stainless steel at 455 to 615 °F, *by H. L. Logan, M. J. McBee, and M. Romanoff, ASTM Mater. Res. Stds.* **3** *(Aug. 1963).*

Photomicrograph of a section cut from the thin wall of the specially designed specimen used in stress-corrosion experiments. The large crack in the wall section—typical of the cracks found on stressed specimens—occurred at some distance from the site of specimen failure. (Etched electrolytically in 10 percent oxalic acid. × 75.)

705–817 O—63——2



Center: **Mrs. Eva Mosby fills a standard flask intended to contain exactly 10 ml. The wide base prevents tipping.** *Upper right:* **Miss Lettie Keels clocks the "time of delivery" for a buret.** *Center right:* **B. C. Keysar monitors the calibration of a 1-ft^3 gas-volume standard. These standards are used by industry to calibrate gas meter "provers."** *Lower right:* **The amount of water delivered by a 10-ml pipet is measured by Mrs. Jo Lembeck in the calibration of glassware.**

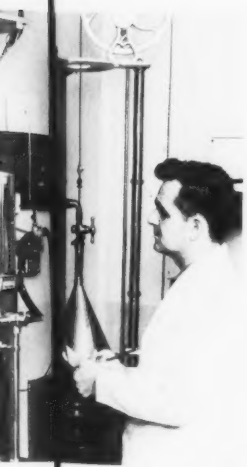


Lower left: **Mrs. Pearl Simmons demonstrates the first step in the calibration of burets by filling a 25-ml instrument with distilled water.** *Center left:* **The temperature of the distilled water used to fill a standard pipet is noted by Mrs. Pauline Rosado.** *Upper left:* **On the scale at right is a 5-gal metal volumetric standard which is being calibrated by Louis Preston.**

Volumetric Measurements

at the National Bureau of Standards

twelfth of a series on NBS measurement and calibration services*



THE BUREAU—as part of its work in maintaining a uniform system of measurement throughout the country—has a comprehensive program for the calibration of volume-measuring equipment. The service provides standards for other standards laboratories, for state weights-and-measures officials who regulate the exchange of goods and commodities within their States, and for industry. Among those who rely on the service are laboratory workers who measure toxin and serum dilutions and red and white blood cell concentrations, or who analyze the content of medicines, cosmetics, and new chemical products.

Volumetric standards may be 5-gal metal measures for inspecting gasoline pumps, or larger measures for testing the meters on vehicle tanks that transport milk or deliver liquid fuel to homes and to service stations. Or they may be glass apparatus for transferring fluids, or flasks for measuring the volume capacity of lubricating oil containers or of the graduates used by the pharmacist in filling prescriptions. They may also consist of gas-volume standards used to calibrate gas meter "provers" or liquefied petroleum gas meters.[1]

Metal Volumetric Standards

The majority of metal standards of liquid volume received for calibration are in units of a gallon, which by definition is 231 in.[3] The units with their equivalents are given at the top of p. 184.

**Previous articles have been:* Calibration of gage blocks, *Feb. 1961;* Thermocouple calibrations, *Mar. 1961;* Calibration of platinum resistance thermometers, *Apr. 1961;* Calibration of inductive voltage dividers, *May 1961;* X- and gamma-ray calibration, *July 1961;* Calibration of microphones, *Nov. 1961;* Calibration of optical pyrometers, *Nov. 1961;* Calibration of flowmeters for liquid hydrocarbons, *Feb. 1962;* Calibration of neutron sources, *Mar. 1962;* Precision resistance measurement, *Feb. 1963;* and A-c voltage calibrations, *Aug. 1963.*

1 liquid pint = 4 gills
1 liquid quart = 2 liquid pints
1 gallon = 4 quarts

A few metal standards in this country are made in metric units. The Bureau uses the liter, which is approximately equivalent to 1000.028 cm^3 or 1.05672 quart or 61.0254 $in.^3$, as the basis for these calibrations.

The most accurate metal standards are those in which the capacity is limited by a glass top called a "slicker plate." They come in both conical and cylindrical shapes, the latter having a small neck to hold the slicker plate, and their nominal capacity is engraved on the outside wall. Another type, designed to hold volumes from a quart to many gallons, is the graduated-neck liquid measure. This standard has a long neck on which is mounted a glass gage and a graduated scale indicating the quantity of liquid it holds. All metal standards carry the manufacturer's name or trademark and a serial number.

The slicker-plate standards received at the Bureau for calibration are evaluated by a weighing procedure. In this procedure, distilled water is added to raise the level above the top, and the slicker plate is pushed across the top to cut off excess water which is then removed from the outside. When the measure is placed on one pan of a balance of adequate size, a tare is adjusted on the other pan until the balance is in equilibrium. The measure is then removed and allowed to drain, after which it is righted, the slicker plate is replaced, and the assembly returned to the balance pan. The weight which must be added to this pan to restore equilibrium is the weight of the drained distilled water.

This weight is converted to volume by the use of Bureau-computed density data that include precise corrections for temperature, atmospheric pressure, and humidity. If the actual volume delivered meets established tolerances, an identifying number is applied to the measure. When the calibrated standard is returned to the manufacturer or user, it is accompanied by a calibration report which lists actual volume and environmental conditions of calibration.

Graduated-neck standards up to 10 gal in nominal size are usually calibrated in a similar weighing process. However, larger measures are calibrated by means of standard measures whose volume is precisely known. A report of calibration is also issued for graduated-neck standards if they meet established tolerances.

Certain requirements are specified for gas-volume standards sent to the Bureau for calibration.[2] For instance, valves and cocks must be leakproof and operate smoothly; volume-adjusting devices must be provided with a means for locking into place; and the maker's name or trademark, serial number, and the nominal capacity of the standard must be engraved on the outside wall.

Portable standards of this type consist of a cylindrical tank called a bell having a nominal capacity of 1 ft^3, together with a slightly larger tank containing a sealing liquid in which the bell is immersed for calibration. The calibration is accomplished by means of a similar tank and a gas-filled standard immersion bottle having a volume of exactly 1 ft^3. When the two assemblies are connected through a series of tubes and flexible hoses and the immersion bottle is slowly and mechanically lowered into its tank, the gas passes from the bottle into the bell as it slowly rises in its tank.

Another type of gas-volume standard designed to deliver 1/10 ft^3 and used for testing small water-sealed rotating-drum meters known as wet-test meters is received at the Bureau for calibration. This calibration is carried out by a weighing procedure in which the standard is filled with water, and the water is then emptied into a container. The weight of the water is measured and converted to the internal volume of the standard.

Arrangements can be made for calibration of other types of metal standards designed for special purposes. Typical of these is a portable standard for testing liquefied gas meters such as those used in the measurement of propane or butane gases. The standard, built to withstand pressures up to 300 $lb/in.^2$, is evaluated in terms of volumetric and pressure data and known compressibility factors. So far, 32 such standards have been calibrated for State use and for industrial gas companies.

Glass Volumetric Standards

Requests for calibration of volumetric glassware are accepted by the Bureau only when circumstances make calibration by NBS facilities necessary. Such glassware includes laboratory pipets, burets, cylindrical graduates, and volumetric flasks.[3] Each instrument bears in permanent legible characters the capacity (usually in milliliters), the temperature at which it is to be used, the method of use—that is, whether it is intended for "containing" or "delivering" a liquid—and, on instruments designed to deliver through an outflow nozzle, the time for completing the delivery process. This last item is included because instruments such as pipets having oversize nozzles empty too quickly and do not give reproducible results. In addition, each instrument bears the name or trademark of the maker and a permanent identification number.

Calibrations are accomplished by technicians who fill the instruments with distilled water and set and read the marks indicating capacity from the lowest point of the liquid meniscus. Volume is then calculated from measurements of the weight of the instrument and of the water contained in it, or from the weight of the water the instrument delivers, depending upon the intended use of the instrument. Corrections are made for volume changes with temperature, so that volume at the temperature inscribed on the instrument can be found.

[1] *For information on fees for NBS calibration services, see NBS Misc. Publ. 250,* Calibration and Test Services of the National Bureau of Standards *(in press). Information on volume measurement fees is also available from the Volumetry Laboratory, National Bureau of Standards, Washington, D.C. 20234.*

[2] Testing of metal volumetric standards, *by J. C. Hughes and B. C. Keysar, NBS Mono. 62, for sale by Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, price 15¢.*

[3] Testing of glass volumetric apparatus, by J. C. Hughes, *NBS Circ. 602, for sale by Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, price 20¢.*

NBS develops methods for determining

THERMAL RADIATION PROPERTIES OF MATERIALS

Working Standards Now Available

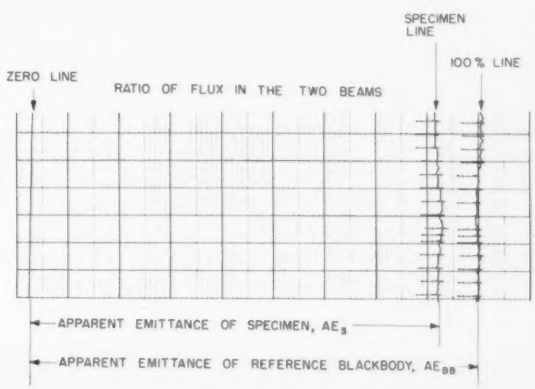
SINCE THE first artificial satellite was put into orbit over 5 years ago, scientists and engineers have had an increasing need for thermal radiation data on materials that might be used for either external or internal parts of space vehicles. To help meet this need, the Bureau, in cooperation with the U.S. Air Force and the National Aeronautics and Space Administration, has developed equipment and techniques for obtaining these data, particularly in the field of thermal emittance. The program, conducted by W. N. Harrison, J. C. Richmond, D. G. Moore, and H. M. Joseph, has also been concerned with measurements on specimens to be used as working standards of thermal emittance. A limited number of these specimens is now available for purchase from the Bureau, for use by other laboratories in calibrating thermal emittance instruments.

The temperatures of spacecraft must be closely controlled, not only for the benefit of living occupants, if present, but also to permit proper functioning of electronic equipment which is regularly carried and which often requires even closer control. Heat transfer by conduction or convection to or from vehicles beyond the atmosphere is negligible because of the high vacuum existing in outer space. Emission and absorption are the mechanisms of heat transfer to and from space vehicles, and they also provide an important mode of heat transfer within the vehicles. Thus the thermal radiation properties of materials composing a spacecraft furnish a key to the control of its internal temperature. These properties include thermal emittance, reflectance, absorptance, and transmittance. The *thermal emittance* of an object is expressed as the ratio of its rate of emission (or radiation) to that of a complete radiator (blackbody) having the same area and temperature, under the same conditions of measurement. The *reflectance* of a particular object is defined as the fraction of incident radiant energy that is reflected, *absorptance* as the fraction that is absorbed, and *transmittance* as the fraction transmitted. The spectral absorptance (the absorptance of the radiation within a narrow wavelength band centered about a given wavelength) is equal to the spectral emittance at the same wavelength. For most applications, thermal radiation properties can be adequately expressed in terms of a series of spectral emittance values for all wavelengths of interest.

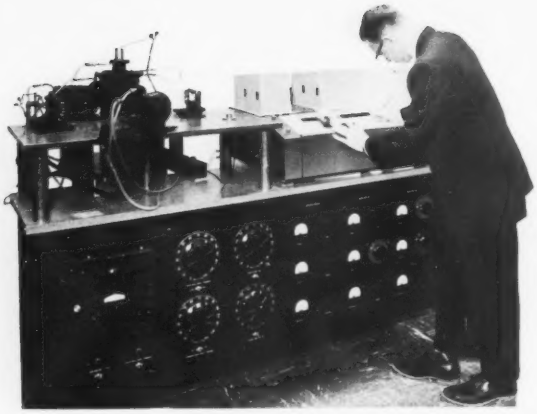
A satellite receives most of its heat by absorption of solar radiation augmented by a much smaller amount which impinges after being reflected from the earth. The portion of the incident solar radiation that is absorbed will raise the temperature of the absorbing object, which, as its temperature increases, will emit radiant energy at an accelerating rate until it reaches a temperature at which it is radiating energy at the same rate it is absorbing it. But this steady temperature differs from one material to another. Solar radiation is almost entirely (97 percent) within the wavelength limits 0.3 to 3.0μ, and the energy emitted by satellites (or other objects) lies principally within the wavelength range 5 to 40μ (corresponding to their own temperature, which is much lower than that of the sun). It follows that the temperature at which a balance is reached can be controlled by selecting a material which must reach the predetermined temperature in order to emit radiant energy at the same rate it absorbs solar energy.

Emittance Determinations

Work at the Bureau has involved three combinations of geometric and spectral conditions for measuring emittance: (1) total hemispherical emittance, for which an overall emittance value, covering all wavelengths and all directions from a surface, is obtained; (2) normal spectral emittance, for which the emittance normal to the specimen, and within a narrow wavelength band centered on a specified wavelength, is measured and


SPECIMEN LINE
100% LINE
ZERO LINE
RATIO OF FLUX IN THE TWO BEAMS
APPARENT EMITTANCE OF SPECIMEN, AE_s
APPARENT EMITTANCE OF REFERENCE BLACKBODY, AE_{BB}

A double-beam ratio-recording spectrometer has been adapted for making normal spectral emittance determinations. Here a section of the recorded chart is shown, containing the "100 percent line," "specimen line," and "zero line." Emittance is computed as the ratio of the apparent emittance of the specimen to the apparent emittance of the reference blackbody furnace.



One facility for measuring the total normal emittance of nonmetallic specimens employs a rotating specimen furnace (*top left*) to reduce temperature gradients within the specimen. Howard Clark inspects a chart containing emittance measurements recorded by a spectrometer (*right*).

plotted as a function of wavelength; and (3) total normal emittance, which also can be computed from spectral normal emittances for all pertinent wavelengths.

In general, the temperature of the specimen and its nature (dielectric or conductor) determine the choice of test method. The program of study involving emittance measurements was divided into four temperature ranges: (1) the vicinity of room temperature (about 300 °K), for making reflectance measurements; (2) 800 to 1400 °K, for normal spectral emittance measurements; (3) 1400 to 1800 °K, for both normal spectral and total normal emittance measurements; and (4) above 1800 °K, for total normal emittance measurements.

Total Hemispherical Emittance Measurements

Total hemispherical emittance measurements are are made by calorimetric procedures.[1] In the Bureau's method for evaluating total hemispherical emittance, a metallic strip specimen is heated in a vacuum chamber approximating a totally absorbing (blackbody) cavity by passing a current through the strip. Total hemispherical emittance is computed from the power input and from the temperature of a measured portion of the strip specimen and the temperature of the surrounding chamber. Total hemispherical emittance measurements of a number of coated and uncoated metallic specimens having various types of surface finishes have been made. In general, the surface finish has a large influence upon the thermal emittance of a specimen.

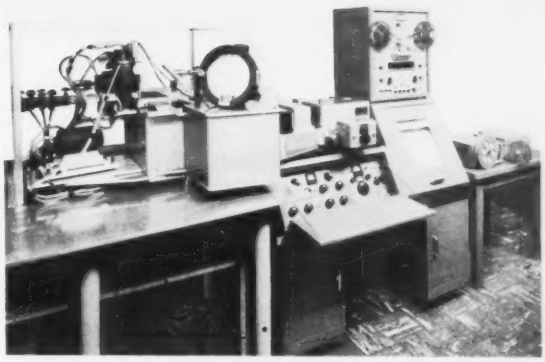
Normal Spectral Emittance

The most urgent need for emittance measurements has been in the 800 to 1400 °K range, where most of the research to date has been carried out. At the Bureau, continuous curves representing spectral emittance at wavelengths from 1 to 15 μ have been made on numerous specimens by means of a double-beam ratio-recording spectrometer.[2] The specimen, heated by passing a current through it, serves as a source for one beam, and a laboratory blackbody furnace, heated to the same temperature, serves as a source for the second beam. The ratio of the radiant flux in the two beams is recorded by the instrument as the normal spectral emittance of the specimen. Two errors exist in the recorded ratio: the "100 percent line" error, arising as a result of differential absorption along the two paths in the spectrometer; and the "zero line" error, resulting from scattering of the reference beam in the spectrometer. Both errors are nearly eliminated by means of calibration curves.

A data-processing attachment has been designed and installed to operate with the spectrometer assembly (1) to produce a corrected graphical record, free from the 100-percent and zero-line errors, for the normal emittance of a specimen as a function of wavelength; (2) to record in digital form on punched paper tape the corrected spectral emittance values, for possible use in separate electronic digital computers; and (3) to accumulate the digitized emittances at wavelengths which have been preselected to yield specific information such as total normal emittance, or absorptance for earth radiation.

Working Standards. Standard specimens have been prepared for use at three levels of emittance and consist of three different materials: (1) polished platinum for low emittance; (2) sandblasted and oxidized Kanthal (an iron, chromium, aluminum alloy) for intermediate emittance; and (3) sandblasted and oxidized Inconel (a nickel, chromium, iron alloy) for high emittance. Each of the three materials was prepared as $\frac{1}{4}\times 8$-, $\frac{3}{4}\times 10$-, and 1×10-in. strips; 2×2-in. squares; and $\frac{7}{8}$-, $1\frac{1}{8}$-, and $1\frac{1}{4}$-in. diam disks. All specimens of a single material were cut from the same sheet of metal and, during their preparation, were treated as nearly alike as possible. Thermal emittance measurements were made only on the $\frac{1}{4}\times 8$-in. strip specimens, at each of 100 wavelengths in the 1 to 15 μ range, at temperatures of approximately 800, 1100, and 1400 °K. These working standards have a stability in total hemispherical emittance to within 0.02 on heating in air at the maximum temperature at which they were calibrated for periods up to 200 hr.

Statistical analysis of the data obtained on three measurements of six specimens of each material at three different temperatures indicates that the overall precision of measurement, expressed as an average standard deviation, varied in emittance from less than 0.005 for the platinum specimens to about 0.009 for



Double-beam ratio-recording spectrometer equipped with a data-processing attachment as an improvement in the means for measuring normal spectral emittance. The spectrometer (*left*) feeds information to the data-processing attachment (*right*), which corrects the graphical record of specimen emittance, records the corrected data on punched paper tape (*extreme right*), and accumulates digitized data on emittance of the specimens at preselected wavelengths.

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the oxidized Inconel specimens. The average standard deviations due to differences between specimens of the same material varied from about 0.007 for platinum to about 0.027 for oxidized Inconel.

The working standards are available from the Bureau at a price of $175 for each oxidized Kanthal or Inconel specimen, and $175 plus $4 per gram for each platinum specimen. The per gram charge approximates $46 per square inch of specimen. Emittance data at 100 wavelengths between 1 and 15μ are enclosed with each specimen. An instruction sheet for handling and caring for the specimen to prevent its contamination is also included. Orders for the specimen should be sent to:

National Bureau of Standards
Attn: Building Research Division
Washington, D.C. 20234

Measurements Above 1400 °K

Because of extremely high temperatures occurring in rocket nose cones reentering the atmosphere, in satellite heat shields, in rocket nozzles, and in future propulsion devices such as the atomic rocket and ion jets, NBS scientists are developing instruments and procedures for evaluating thermal emission characteristics of materials at temperatures above 1400 °K. Most conventional metals cannot be used in this temperature region, and most refractory metals not only are difficult to fabricate but also have poor oxidation resistance. Because refractory ceramic oxides will withstand these high temperatures, they are potentially useful for such applications.

Nonmetals generally exhibit appreciable thermal gradients when tested at high temperatures because they have a low thermal conductivity and a relatively high thermal emittance. The radiant energy emitted from most nonmetals does not originate in a microscopically thin surface layer, as it does in the case of metals (radiant energy from lower levels being reabsorbed), but instead significant amounts of radiant energy are transmitted outward from points of origin appreciably below the surface. This condition increases the difficulty of making accurate thermal emittance determinations.

Because of differences in experimental problems, separate methods were selected for measuring emittance at temperatures below and above 1800 °K. At the lower temperatures, a hollow cylindrical specimen is rotated in a furnace that is equipped with a water-cooled viewing port. Because the specimen is rotating, freshly heated areas arrive continuously at the port. When the specimen reaches the desired temperature, measurements are made with a spectrometer. Tests conducted thus far with alumina specimens show that temperature gradients have been reduced to workable levels. A heat transfer analysis has shown that with the specimen rotating at 100 rpm its temperature can be measured with sufficient accuracy by a thermocouple placed within the central core of the specimen.

For total normal emittance measurements at temperatures between 1800 and 2500 °K, specimens consisting of small flat disks are employed.[3] A shallow hole is drilled at the center of each disk to serve as a reference cavity. The specimens are heated in refractory metal crucibles by induction, and the temperature is measured by an optical pyrometer. Data from a vacuum thermocouple detector, mounted to receive the radiant flux, are amplified and recorded. The shallow holes do not radiate as blackbodies, but the shallow depth facilitates minimizing temperature gradients; and when the dimensions are known accurately, a correction to blackbody conditions can be computed. Further work is necessary, however, to establish the limits of accuracy of this method.

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[1] Total hemispherical emittance of coated and uncoated Inconel and types 321 and 430 stainless steel, *by J. C. Richmond and W. N. Harrison. J. Res. NBS* **66C**, *No. 3 (Eng. and Instr.), 261 (July–Sept. 1962).*

[2] An approach to thermal emittance standards, *by J. C. Richmond, W. N. Harrison, and F. J. Shorten. Proc. Symp. Measurement of Thermal Radiation Properties of Solids, NASA Tech. Publ. TP 31 (to be published).*

[3] The use of shallow-hole reference cavities for high temperature emittance measurements, *by D. G. Moore. Proc. Symp. Measurement of Thermal Radiation Properties of Solids, NASA Tech. Publ. TP 31 (to be published). Also* Thermal Radiation Symposium, *NBS Tech. News Bull.* **46**, *181 (Dec. 1962).*

ANALOG COMPUTER

for plasma thermometry

A SIMPLE, inexpensive analog computer for the rapid determination of the radial characteristics of inhomogeneous arcs and flames has been developed by J. B. Shumaker, Jr., and C. R. Yokley of the temperature physics laboratory. This computer,[1] accurate to within 5 percent, provides radially resolved spectral data from which temperature, electron density, and transition probabilities can often be calculated. Its use, which permits the almost instantaneous recording of the spectrum at any four preselected radial positions in the source, makes it as easy and rapid to study inhomogeneous as homogeneous sources.

Spectroscopic studies of arcs and flames are often complicated by the fact that observations made across the radius of the source include contributions from the outer, cooler regions as well as from the hot core. The contributions of the various radial zones can be determined through numerical solution of Abel's integral equation, but this is a laborious and time-consuming process. Digital computers can be used to perform the necessary calculations, but they are expensive, and the data must be put in a format, such as punched cards, acceptable for machine input. Therefore, an analog computer was developed which, operating directly on the photomultiplier output resulting from scanning the source, provides radially resolved spectra immediately.

The computer is presently being used in conjunction with studies of a wall-stabilized, high-current plasma arc.[2] This cylindrical arc is viewed "side-on"; that is, in a radial direction at right angles to its axis. The arc is imaged by an optical system through a 12-sided prism onto the slit of a spectrometer. The prism is rotated at exactly 500 rpm so that the spectrometer is presented with an edge-to-edge traverse of the arc at 100 c/s, a rate compatible with the characteristics of filters included in the computer circuitry. The 100-c/s signal from the photomultiplier tube at the spectrometer exit slit is Fourier analyzed while the spectrometer scans slowly in wavelength. The computer then weights and sums the Fourier amplitudes, producing a recorder trace of the source intensity versus wavelength at the four radial positions.

The Abel equation on which the computer operates is

$$F(x)=2\int_x^R \frac{G(r)rdr}{\sqrt{r^2-x^2}}$$

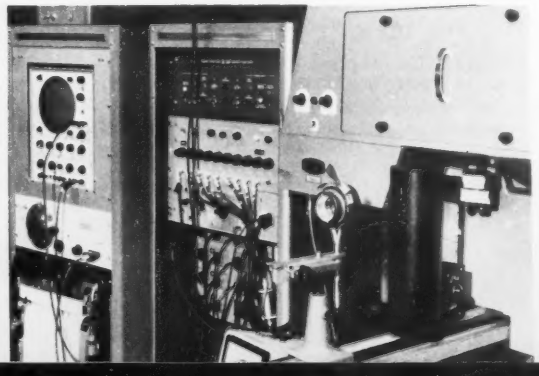
and its solution

$$G(r)=-\frac{1}{\pi}\int_r^R \frac{\frac{dF(x)}{dx}\,dx}{\sqrt{x^2-r^2}},$$

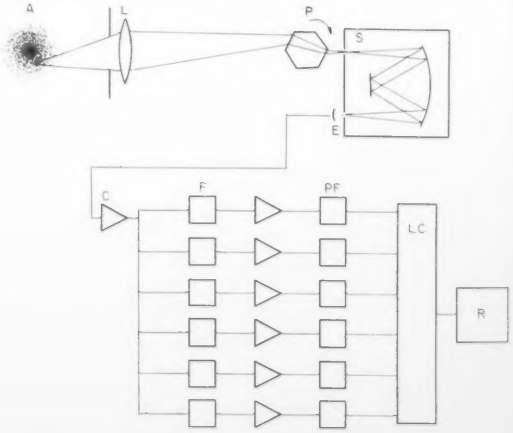
where $F(x)$ is the side-on arc profile and $G(r)$ is the true radial distribution of intensity.

The computer uses a six-term Fourier expansion to approximate $F(x)$, employing cosine functions as the arc is symmetric around its center. The position in the source for which a solution is generated is determined by the value of a set of plug-in resistors, a different set being used for each desired position. Thus, by merely changing a small component, the spectrum at any preselected radial distance can be determined.

The analog computer developed for determining the radial characteristics of inhomogeneous spectral sources is in the two racks at left. This computer produces—almost immediately—a strip-chart record of the spectral intensity at any preselected radial position in the source. The radial position for which the computer makes an analysis is controlled by simple plug-in banks of resistors.



An analog computer for the rapid radial characterization of inhomogeneous arcs and flames. Light from source *A* passes through lens *L* and rotating prism *P* and is presented to spectrometer *S* at the rate of 100 c/s. The output of photomultiplier *E* passes through amplifier *C*, filters and amplitude and phase-correcting amplifiers *F*, peak followers *PF*, linear combiner *LC*, and the computer results are recorded on strip chart *R*.



TS

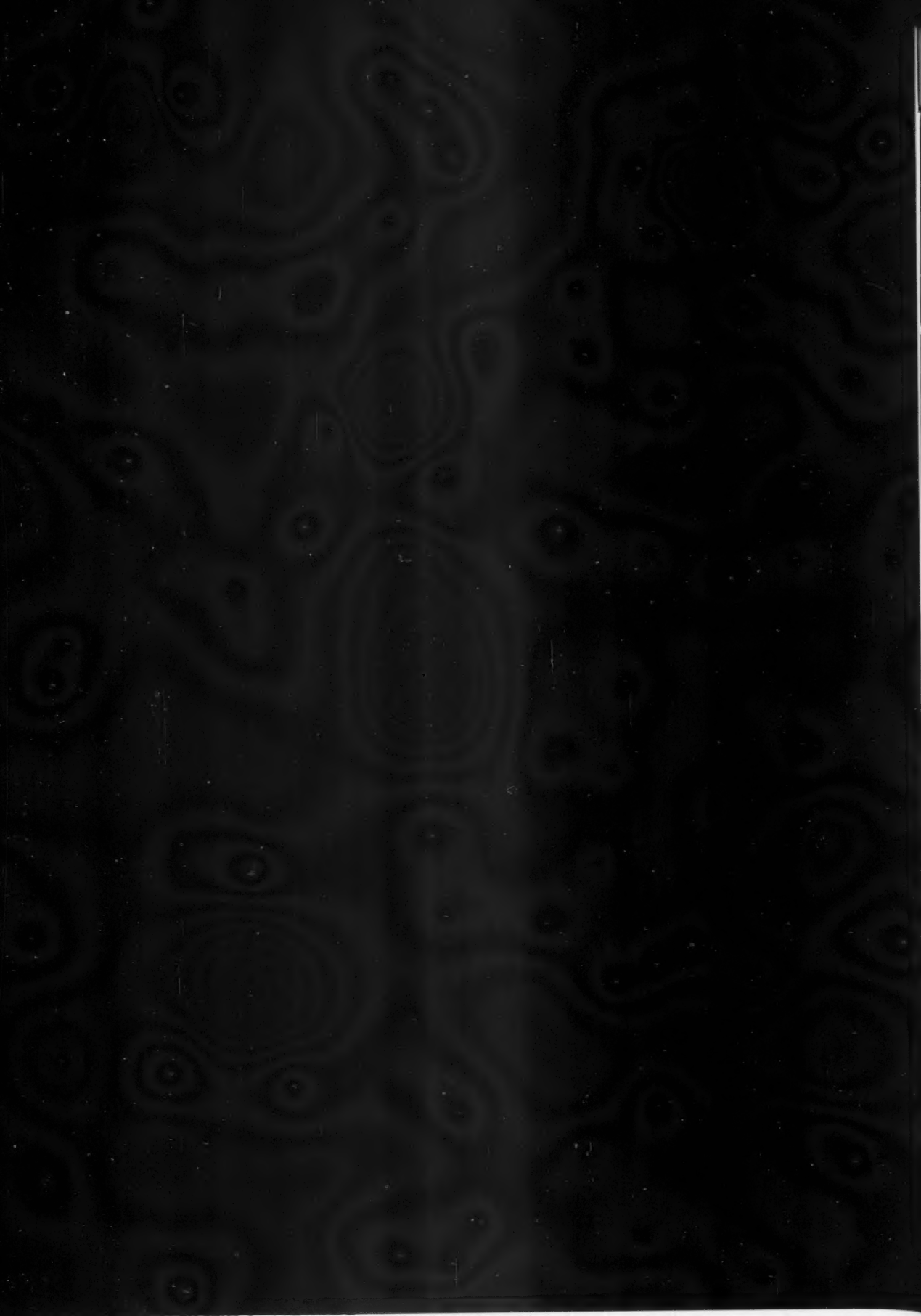
ERRATA
for
NATIONAL BUREAU OF STANDARDS TECHNICAL NEWS BULLETIN
October 1963, Volume 47, Number 10

In the article New Values for the Physical Constants, Table 2, page 176, Adjusted values of constants, note the following corrections:

Item	Now reads in part	Should read
Planck constant	2nd line under Symbol now reads h	$\hbar$
First radiation constant ($2\pi hc^2$)	Système International unit now reads 10^{-8}	10^{-16}

The correction to the first radiation constant also applies to the wallet-size card mentioned in footnote 1 (p. 177).

November 8, 1963.



The computer is constructed largely of inexpensive, commercially available components; the filters and sign-sensing relay drive elements are the only components requiring special design. The entire computer, including the power supply for the photomultiplier, and a strip chart recorder, is contained in two 6-ft racks.

The system is calibrated through the use of a tungsten strip lamp in place of the arc. The d-c photocurrent is measured at each wavelength of interest while the prism is stationary, and then a 100-c/s sinusoidal current is applied to the computer, the recorder output providing a calibration of the electronics.

The performance of the system was checked by determining the transition probability of the NI 4935–4915 multiplet with the computer and by conventional numerical techniques. The spectra were provided by exciting nitrogen and mixtures of nitrogen and 1 percent ammonia in a high-current arc. The transition probabilities as determined by the computer compared favorably with those computed numerically, as shown below:

Wavelength	*Analog computer result*	*Numerically computed result*
4935 Å	1.66×10^{6}	1.73×10^{6}
4915 Å	0.75×10^{6}	0.77×10^{6}

In applications where the differences between the two methods are acceptable, the analog technique will provide a useful and timesaving means of plasma diagnostics.

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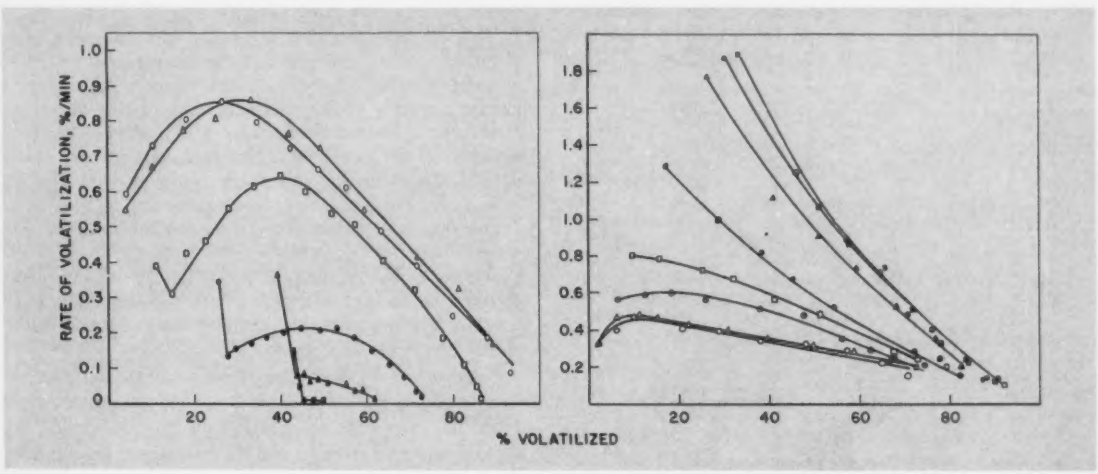
[1] *For further details, see* The use of an analog computer in side-on arc spectroscopy, *by J. B. Shumaker, Jr., and C. R. Yokley, Applied Optics (in press), and* A computer for the Abel Inversion, *Rev. Sci. Instr.* **34**, *551 (May 1963).*

[2] Arc source for high temperature gas studies, *by J. B. Shumaker, Jr., Rev. Sci. Instr.* **32**, *65 (1961).*

Pyrolysis of Polytrifluoroethylene

RESEARCH on the degradation of polymers exposed to high-energy radiation preceding thermal treatment should provide information useful in estimating their resistance to outer space environment. Scientists at the Bureau, in work sponsored by the National Aeronautics and Space Administration, recently studied the thermal decomposition of polytrifluoroethylene which previously had been exposed to gamma radiation or to alkali treatments. Results obtained by Sidney Straus and Leo A. Wall show that both exposures introduce crosslinkage in the polymer chain.[1] Crosslinkage resulting from preirradiation involves primarily branched structures that are less stable than the original polymer structures; the alkali treatment leads to a crosslinked main chain that decomposes to give a very stable residue.



Left: **Effect of prior alkali treatment on the rate of volatilization at 390 °C of polytrifluoroethylene. The alkali treatment prevented the polymer from volatilizing to 100 percent, and produced a very stable residue; the amount of residue increased with higher amounts of alkali. This graph shows that the rate of volatilization of the polymer decreased as the polymer was treated with greater amounts of alkali. Ratio of polymer to alkali used: ○—untreated polymer; △—4 g/0.1 g; □—4 g/0.5 g; ●—4 g/1 g; ▲—4 g/2 g; ■—4 g/4 g.**

Right: **A major effect of prior gamma radiation on the rate of volatilization at 380 °C of polytrifluoroethylene is increased thermal degradation of the polymer with increased radiation dose, even though crosslinking is also introduced. This graph shows (in ev/g$\times10^{-20}$) how the rate of volatilization of the polymer increased with radiation dose. ○—untreated polymer; △—0.51; ⊙—5.1; □—9.44; ●—19.38; ▲—43.86; ◒—63.24; ■—103.53.**

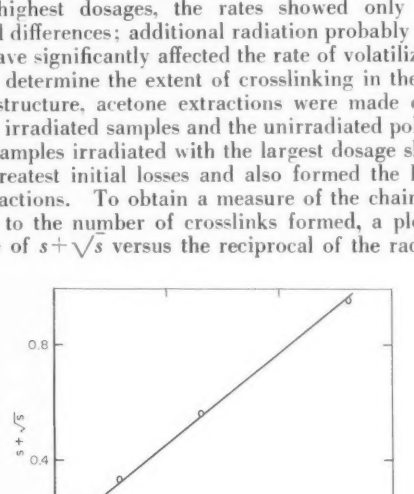
Gamma Radiation

The polytrifluoroethylene prepared at NBS was a white, powdery solid having a molecular weight in the range of 100,000 to 500,000. The polymer powder was compressed into a smooth plastic tape approximately 8 to 10 mils thick. Strips cut from the tape were sealed in Pyrex tubes and the tubes were irradiated by a cobalt 60 source to doses of from 1 to 203 megaroentgens (Mr). These strips absorbed 0.51×10^{20} electron volts per gram (ev/g) when given a dose of 1 Mr.

The treated polymers were then pyrolyzed in an evacuated glass tube apparatus to determine the products of decomposition by mass spectrometry and in a tungsten-spring balance apparatus to determine the rates of degradation. Initial rates of volatilization at specific temperatures were calculated and plotted; certain aspects of the thermal decomposition mechanism could be postulated from the shape of the resulting curves and from the composition of the degradation products.

The tungsten-spring balance experiments at 380 °C with seven irradiated samples (doses of 0.51, 5.0, 9.44, 19.38, 43.86, 63.24, and 103.53 ev/g$\times 10^{-20}$) and with an unirradiated sample indicated that volatilization rates increase decidedly with radiation dose. At the two highest dosages, the rates showed only slight initial differences; additional radiation probably would not have significantly affected the rate of volatilization.

To determine the extent of crosslinking in the polymer structure, acetone extractions were made on the seven irradiated samples and the unirradiated polymer. The samples irradiated with the largest dosage showed the greatest initial losses and also formed the largest sol fractions. To obtain a measure of the chain fractures to the number of crosslinks formed, a plot was made of $s+\sqrt{s}$ versus the reciprocal of the radiation dose, where s is the sol fraction. The slope intercept on the Y-axis, denoting the ratio of scissions to crosslinks, was 0.14, indicating that approximately seven crosslinks were formed for each chain fracture.



Prior gamma irradiation of polytrifluoroethylene introduced crosslinking and caused increased degradation of the polymer on heating. To obtain a measure of the chain fractures to the number of crosslinks formed, this graph was made of $s+\sqrt{s}$ versus the reciprocal of the radiation dose $1/r$, where s is the sol fraction. The slope intercept on the Y-axis denotes the ratio of scissions to crosslinks. The intercept was 0.14, indicating that for each chain fracture approximately seven crosslinks were formed.

These studies demonstrate that major effects of gamma radiation on polytrifluoroethylene are both crosslinking and degradation (main chain scission) occurring at random on heating. Moreover, both are proportional to the radiation dose. In spite of the fact that crosslinking was introduced, the polymer decomposed more readily on heating as the radiation dose was increased. This effect suggests the operation of a crosslinking mechanism producing primarily branched structures that are not as stable as the original chain structure. Polymer samples subjected to very large radiation doses should break down readily into low molecular weight oils when given careful heat treatment.

Alkali Treatment

For polymers used as electrical or thermal insulators required to operate at elevated temperatures, it is desirable to have materials that will, even if changed in chemical structure, leave a substantial amount of nonconducting residue. To examine the effect on the rate of degradation, and possibly to activate the polymer toward thermal dehydrofluorination favoring the formation of an inert residue, powdered samples of the unirradiated polymer were heated at 50 °C with varying percentages of sodium hydroxide for 3 days. The reaction products were washed to remove the alkali and they were then (1) studied for rates of volatilization at 390 °C in the tungsten-spring balance, and (2) pyrolyzed in each case at 500 °C for 1 hr in the tube apparatus, so that the volatile products and the residues could be collected and weighed.

The alkali treatment prevented the polymer from volatilizing to 100 percent; furthermore, increased amounts of alkali increased the residue. Mass spectrometric analysis of the products showed that the alkali treatment promoted the formation of hydrogen fluoride, which is easily removed by heating the polymer at elevated temperatures. This ready removal thermally stabilizes the polymer, and accelerates the formation of double bonds which facilitate crosslink production. The crosslinked main structure then decomposes quite slowly at the same temperature; the greater the alkali content, the slower the rate. Although the polymer is actually made less stable initially by the treatment, it leaves a very stable residue.

Further pyrolysis studies of the alkali-treated samples were made in the tube apparatus at temperatures up to 1000 °C. Data on samples heated for 10 min at 1000 °C indicate an extremely stable residue for the samples. It could be inferred by extrapolation that the residue remaining at 1000 °C would resist temperatures up to the region of 2000 or 3000 °C.

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[1] *For further technical details, see* Pyrolysis of polytrifluoroethylene: Influence of gamma radiation and alkali treatment, *by Sidney Straus and Leo A. Wall, Soc. Plastics Engrs. Trans.* **3**, *No. 4, Oct. 1963.*

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